

Characterization of Organic Materials Using High-Resolution Synchrotron Diffraction

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Beamline(s): **X3B1**

Introduction: Structure elucidation of organic materials utilized in the photographic industry is required to understand materials properties and processing. Though many of these organic compounds are crystalline, the ability to grow single crystals large enough for single-crystal diffraction experiments is often impossible. Powder X-ray diffraction techniques are excellent for fingerprinting these organic materials, but conventional diffraction patterns often lack the necessary peak resolution required for unit-cell indexing. This resolution issue is significant especially for materials that have low symmetry and large unit cells. X-ray diffraction experiments using synchrotron radiation have been performed to obtain high-resolution diffraction patterns for the purpose of determining the crystal structure of selected organic chemicals.

Methods and Materials: The materials analyzed in this study are proprietary organic compounds, all utilized in photographic products. Samples for X-ray analysis were initially characterized using a laboratory X-ray diffraction source (Rigaku rotating anode, CuK α radiation, Bragg-Brentano geometry, diffracted beam monochromator). Acceptable materials were then loaded into 1mm glass capillaries for data collection on the high-resolution diffractometer on the X3B1 beamline ($\lambda = 0.699927$ Å or 1.149957 Å). Synchrotron data collection experiments were performed with either a diffracted beam analyzer crystal or a 0.03° diffracted beam sller slit collimator.

Results: High-resolution diffraction patterns were collected for five materials. In all of the samples analyzed, the synchrotron diffraction data sets were found to be superior in peak resolution when compared to the laboratory source data. As shown in Figure 1, the selected range diffraction patterns demonstrate the advantage of the synchrotron-based X3B1 diffractometer compared to the laboratory diffractometer. Attempts to index the diffraction patterns obtained from our laboratory proved to be unsuccessful since peak position could not be accurately defined for all peaks. The well-resolved peaks obtained using the synchrotron diffractometer significantly enhance the possibility to obtain a correct unit-cell and indexing of the diffraction peaks observed in the diffractogram. For the sample analyzed in Figure 1, the unit cell was found to be Monoclinic, $a = 4.7947$ Å, $b = 37.8571$ Å, $c = 14.7483$ Å, $\beta = 90.529^\circ$. Unit cells and indexing have also been determined for two of the remaining four organic materials in this study. Structure elucidation has been completed on one of these chemicals and work continues on the remaining four.

Conclusions: In this study, high-resolution diffraction data have been successfully collected from organic materials. The ability to cleanly resolve closely spaced diffraction peaks has proven to be critical for successful unit-cell indexing and structure elucidation.

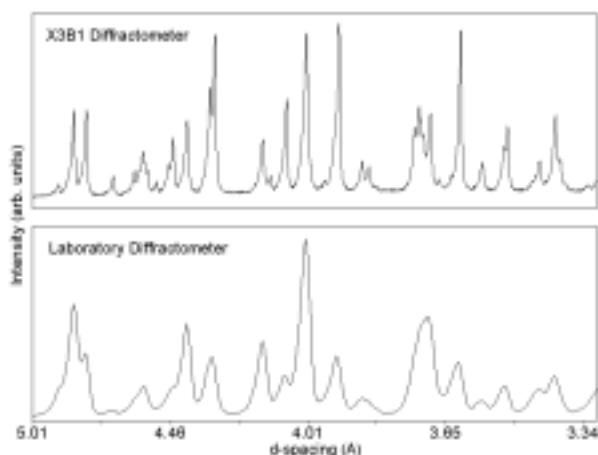


Figure 1. Selected range X3B1 versus Laboratory X-ray diffraction patterns for an organic material analyzed in this study. (Plotted as d-spacing (Å) on the X-axis to allow direct comparison of the two data sets collected with two different radiation wavelengths).